RESEARCH ARTICLE

A Comparison of Physico-Chemical Properties of Neem and Karanja Oil Methyl Esters

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ABSTRACT: Present work reports an optimized protocol for the production of biodiesel (methyl ester) through two stage process i.e., esterification and transesterification of Neem and Karanja oils. The effect of different parameters on the production of Neem and Karanja oil methyl esters were studied. The methyl ester with best yield and quality was produced in case of Neem oil Methyl Ester at 9:1 mole ratio and 1.5 wt.% of catalyst KOH the yield was 90% and Karanja oil Methyl Ester at 9:1 molar ratio and 1 wt.% of KOH catalyst the yield was 92%. It was noted that greater or lower the concentration of KOH or methanol than the optimal values, the reaction either did not fully occur or lead to soap formation. The quality of the two methyl esters was analyzed by using Gas Chromotography – Mass Spectroscopy (GC-MS), Elemental analyzer and Fourier Transform- Infrared Spectroscopy (FTIR) and was compared with each other. CHNOS are found by using elemental analyzer and are compared with the biodiesel standards (ASTM D6751). Physico-chemical properties of the methyl esters were compared to that of petroleum diesel. The produced biodiesel was found to exhibit fuel properties within the limits prescribed by the American Standards for Testing Material (ASTM D6751). It was concluded that Karanja oil was more suitable than Neem oil for the production of methyl ester.

KEYWORDS: Biodiesel, Transesterification, Neem oil, Karanja oil, GC-MS, Elemental Analyzer, FTIR.

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1. INTRODUCTION

The major sources of the world's energy needs are petroleum, coal and natural gases which are fossil-derived and non renewable. The world, at large, depends on petroleum as the energy source for the transportation sector. The life of convectional fossil fuels has become limited in the present era, where the use of energy is growing fast.

On the global scale, increase in the emission rates of greenhouse gases produced from the use of these conventional fossil fuels presents a threat to the world climate. The major exhaust emissions that contribute to pollution and global warming are SO_2 and CO_2. It has been shown that 98% of carbon emissions result from fossil fuel combustion [1,2].

Recently, developing countries such as India and China have experienced a significant increase in energy demand. Moreover, the persistent hike in global prices of crude oil is becoming the major issue in every country. This exacerbates the situation in the form of dwindling production rate, the instabilities in the petroleum production and the processing costs such as; desulphurization in order to meet stringent emission norms etc [3]. This, inevitably, reflects an adverse impact on the local economy of many countries, especially the oil importing countries, by posing a severe burden on their foreign exchange [4]. Therefore, the aforementioned obsessive issues were considered to be the important trigger for many initiatives, to search for the alternative source of energy, which can supplement or replace fossil fuels [5 -7].This study investigates the production of biodiesel from neem and karanja oil, The effect of process parameters—Mole ratio of oil to methyl alcohol, Catalyst concentration, Reaction Temperature, Reaction Time and Process time respectively on biodiesel yield, to evaluate the physical and chemical characteristics of Methyl Ester produced and analyze GC-MS, Elemental Analyzer (CHNOS) and FTIR were studied.

2. MATERIALS AND METHODS

2.1. Experimental Setup

Three necked flat bottomed glass flask of 2000ml capacity was used for transesterification reaction. A double coiled reflux condenser was fitted to a neck of the glass flask for condensing methanol vapors during the reaction. Cool water was circulated through coils of the condenser. A glass thermometer of the range 0 – 360^{o}C was fitted to the second neck of the flask to measure the temperature of the reaction mixture. A plate heater with a magnetic stirrer was used for uniform heating of the contents of the flask. Oil, methanol and other chemicals were transferred through the third neck of the flask at the start of each experimental run using glass funnel and was stoppered during the reaction. A constant temperature bath was provided to maintain the temperature of the contents in the flask in the range 60 – 63 ^{o}C.

2.2. Experimental Procedure

2.2.1. Preconditioning of oil

Preconditioning of oil involved the removal of the moisture and neutralization of free fatty acids (FFA) [8]. Usually the present feed (Used vegetable oil) consist less than 5% of FFA but they need to be neutralized as they result in the formation of soap.

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For determination of FFA present in the oil taken, we titrated the oil sample with 0.1N KOH solution with Phenolphthalein as indicator. Volume in ml of 0.1N KOH required to neutralize 1gram of oil was called acid value, and with this we determined the amount of KOH needed to neutralize the FFA's in the oil. Thus by adding calculated amount of KOH, FFA's were removed in the form of soap.

Removal of moisture involved heating the oil sample up to 105-120^{0}C and maintaining it there for few minutes so that all moisture gets evaporated. If required vacuum pump arrangement was used to remove the moisture present.

2.3 Trans-Esterification Reaction

Transesterification reaction takes place in two stages

***First stage (Acid catalyzed stage*)**

The free fatty acids can be reduced to esters by two processes viz. hydrolysis, methanol with acid catalyst. The treated oil was taken in liters and heated to 35^{o}C to melt the solid fats presented in the oil. Methanol of 99% pure was added (0.1 liters/liter of oil) to the heated oil. It was stirred for five to ten minutes (methanol is a polar compound; oil is strongly non–polar; hence a suspension will form). One milliliter of 95% pure sulfuric acid (H_2SO_4) was added for each liter of oil using a graduated eye dropper. The compound was stirred for one hour maintaining the temperature at 35^{o}C. Heating was stopped and the mixture was stirred for another hour. The mixture was allowed to settle for eight hours in a decanter to remove glycerin and chemical water.

Second stage (Base catalyzed stage)

For each liter of oil/fat 0.12 liter of methanol (12%by volume) was measured to which it is added 6.5 grams / liter of lye (Sodium hydroxide, NAOH). The mixture was stirred thoroughly until it forms sodium methoxide. Half of the prepared sodium methoxide was poured into the unheated mixture and the mixture was stirred for five minutes. This will neutralize the sulfuric acid. The mixture was heated to 55^{o}C and the whole reaction was maintained. Remaining sodium methoxide was added to the heated mixture and stirred at low speed of not more than 500 to 600 rpm. After one hour the mixture was poured into a decanter and allowed it to settle for 8 hours. As glycerin was heavier than the methyl ester, it will settle at the bottom. The glycerin was separated from the methyl ester.

2.4. Influence of process parameters

The experiments were carried out and adopted considering the variables, temperatures (30, 40, 50,55,60,65 and 70^{o}C) and methanol: oil molar ratios (6:1– 12:1), catalyst concentrations (0.5–2 wt%) and reaction time of (30–120) min. All experiments were done in triplicate, and the average data were presented.

2.5. Purification of products

Bubble wash method was used, but there is no need to monitor pH value of the oil any more one milliliter of phosphoric acid (H_3PO_4) is added to the washing water first. One third water by volume to the oil is being used and the oil is bubble washed for two hours. The mixture is allowed to settle in a decanter for one hour and the water is drained off later. The methyl ester is heated to 100^{o}C to dispense with the traces of water and preserved.

2.6. Characterization of methyl ester

Physical properties like Flash Point: Fire Point: Calorific Value: Kinematic Viscosity: Density and Specific Gravity: Iodine value was analyzed using standard procedure[8] .

2.6.1. Chemical properties

The quality of the methyl ester was analyzed by using Gas Chromotography – Mass Spectroscopy (GC-MS), Elemental analyzer and Fourier Transform-Infrared Spectroscopy (FTIR) and is compared with each other. GC-MS and FT-IR analysis were studied to identify and confirm the presence of fatty acid methyl esters. CHNOS are found by using elemental analyzer [9].

3. RESULTS AND DISCUSSIONS

3.1. Effect of Different Parameters on Production of Methyl Ester from Neem and Karanja Oil

3.1.1 Effect of Methanol to Oil Ratio on Methyl Ester Yield

In this experiment, Neem oil was used with varying molar ratios of methanol and oil (6:1, 7:1, 8:1, 9:1, 10:1 and 12:1) with 1 wt.% KOH at 60°C with 90 min reaction time. As mentioned, the transesterification activity also depends on the molar concentrations of methanol to oil. Molar ratio increases from 6:1 to 12:1, methyl esters content decreased methyl ester yield and excess of molar ratio increases much then methyl ester yield also increases. Excess of methanol is required to shift the equilibrium favorably during transesterification for better yields of methyl ester. Therefore, 9:1 molar ratio gives a better result than other molar ratios for Neem oil and also repeated same procedure done for Karanja oil. The highest yield is found with molar ratio 9:1. The higher alcohol molar ratio interferes with the separation of glycerol because there is an increase of solubility. In addition, an excess of alcohol was able increase the conversion of dimonoglycerides, but there is possibility of recombination of esters and glycerol to form monoglycerides because their concentration and also increasing during the course of the reaction, in other words the reactions conducted with low molar ratios. Hence, the alcohol molar/oil ratio is an important factor that must be always optimized. The yields of Neem and Karanja oil methyl ester were shown in Figure 1. (Figure is missing). Similar results were also obtained by Muthukumaran et al., using non-edible Madhuca indica oil for biodiesel production [10].

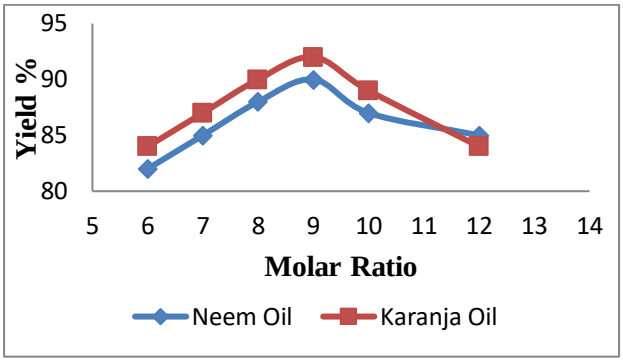

Figure 1: Methyl ester yield with molar ratio

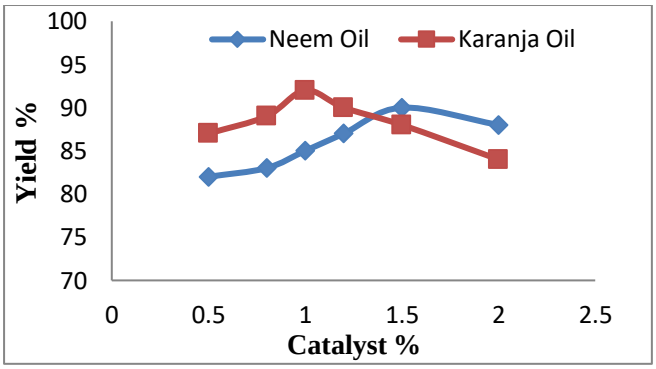

Figure 2: Methyl Ester Yield with Catalyst (KOH) %

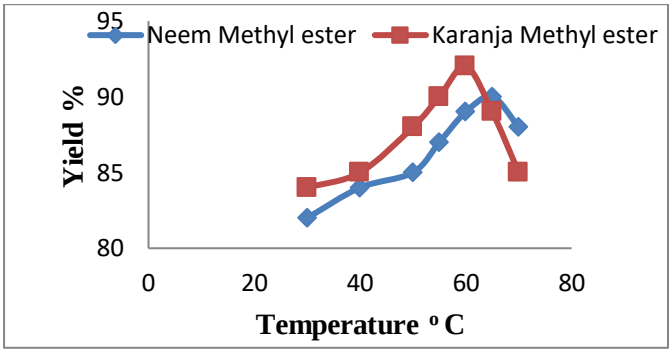

Figure 3: Methyl Ester Yield with Temperature

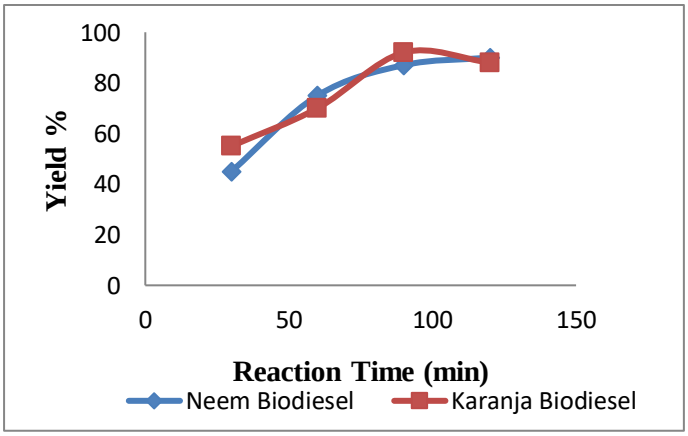

Figure 4: Methyl Ester Yield with Reaction Time

3.1.2 Effect of Catalyst (KOH) weight on Methyl Ester Yield

Generally, catalyst concentration can affect the rate of Methyl ester reaction and so KOH catalyst used was 0.5, 0.8, 1.0, 1.2, 1.5, 2.0 wt.% concentration for Neem and Karanja oil in that, 0.5 wt% shows only 82% Methyl ester yield for Neem oil and 1 wt.% shows only 85% methyl ester yield for Neem oil. For 2 wt.% catalyst shows methyl ester yield to 88% thereby yield decreases due to its higher concentration of catalyst addition gives negative effect of methyl ester production. Whereas describes 1.5 wt% of catalyst shows 90% methyl ester yield for Neem oil higher methyl ester yield than other concentrations. In basic catalyst, a slight decrease in ester content was observed in the experiments with 2 wt. % of catalyst with regard to the experiments with 1.5 wt. %. This is because the addition of excess alkaline catalysts caused more triglycerides participation in the saponification reaction, resulting in increased production of soap and mentioned reduction of the esters yield. So, any increase in concentration of catalyst beyond the neutralization limit results in decrease in methyl ester conversion. Similarly for Karanja oil the optimized concentration was found to be 1.0 wt.%. The yields of Neem and Karanja oil methyl esters, at different concentration of KOH catalyst were shown in Figure 2.

3.1.3 Effect of Temperature on Methyl Ester Yield

The primary advantage of higher temperatures influences to shorter reaction time. However, higher reaction temperatures causes methanol to vaporize resulting in decreased yield. In this process, the temperature used 30, 40, 50, 55, 60, 65 and 70°C in order to optimize Methyl ester yield with using the constant parameters are: alcohols to oil molar ratio of 9:1 with KOH concentration of 1.5 wt. % for Neem oil and 1.0 wt.% for Karanja oil at 90 min reaction time. Thereby the experiment describes methyl ester yield with respect to different temperature for both the oils. 30°C shows methyl ester yield were 82% for Neem oil, again increase the temperature to 400°C and methyl ester yield also increases. Further the temperature increases to 65°C, the methyl ester yield were 90% for Neem oil. When the temperature decreases, the rate of reaction also decreases. Therefore, the equilibrium concentration was strongly conditioned by the temperature and the optimum temperature is 65°C for Neem oil methyl ester process.

Similarly for Karanja oil increase in the temperature of the reaction yield is increasing but after a certain increase in temperature yield is decreased due to non availability of methanol i.e. methanol is vaporizing [11]. The optimum value for Karanja oil was 55°C. The yields of Neem and Karanja oil methyl esters were shown in Figure 3.

3.1.4 Effect of Reaction Time on Methyl Ester Yield

According to many researchers, the methyl ester yields are directly proportional to the reaction times used. This experiment was conducted from the reaction time of 30, 60, 90 and 120 min with the constant parameters: 65°C of reaction temperature, 1.5 wt.% of KOH catalyst used with 9:1 molar ratio of Neem oil and for Karanja Oil is 55°C, 1wt.% KOH catalyst methanol to oil ratio is 9:1.

The experiment values show that effect longer mixing gives higher yield than using shorter time. So, 90 min of reaction time gave a good result than other reaction times used here. However, based on the results, it shows that the methyl ester yields were lower when reaction time of 120 min was used. This undesirable result may be due to the higher soap formation when longer reaction time was used. Thus, the rate of soap formation was also increased. The yields of Neem and Karanja oil methyl esters at 30, 60, 90 and 120 min were shown in Figure 4.

3.2 FFA analysis of Methyl Ester using GC- MS

The fatty acid composition of the oils seems to have an important role in the performance of the methyl ester in diesel engines. Based on the fatty acid composition and many other parameters, the EU biodiesel specifications will be mandatory to limit the oxidative stability, as it may be a crucial parameter for injection pump performances Moreover, the stability of the fuel is a quality parameter established by the ANP- National Petroleum Agency in Brazil, being its evaluation and control necessary.

Vegetable oils are natural products consisting of ester mixtures derived from glycerol (triglyceride), whose chains of fatty acid contain about 14 to 20 carbon atoms with different degrees of unsaturation. The transesterification reaction consists in the conversion of the triglyceride molecules, by means of the action of short chain alcohol, i.e., methanol, ethanol into the corresponding fatty acid esters. According to the source of oil seed, variations in the chemical composition of the vegetable oil are expressed by variations in the molar ratio among different fatty acids in the structure. The prepared Methyl Esters i.e., Neem methyl ester was analyzed by Agilent 6890 series GC-MS and Karanja methyl ester was analyzed by Agilent GC- 5973 N GC-MS system to determine the composition of fatty acids. The methyl ester is mainly formed by transesterification of saturated and monounsaturated fatty acids while the remaining polyunsaturated and some bulk saturated fatty acid are responsible for high viscosity. The higher level of unsaturated fatty acid reduces fuel quality, because of its easy oxidation. The GC-MS results of both the methyl esters were shown in Figure 5 and 6. (Missing)

3.3 Elemental Analysis:

3.3.1 Oxygen and its Calorific Value

In general, the calorific value (or heat of combustion) of methyl ester is less than that of fossil diesel due to presence of C-O and C-C bonds. The fossil diesel is made up of a mixture of various hydrocarbon molecules and contains little oxygen (up to 0.3%) and small amount of sulfur (0.25%), while the methyl ester consists of three basic elements namely: carbon, hydrogen, significant amount of oxygen. The increase of O_2 in methyl ester (biodiesel) is related to the reduction of C and H, causes the lower value of lower calorific value (LCV) of methyl ester as compared to

that of fossil diesel, because O_2 is ballast in fuel and 'C and H' are the sources of thermal energy. Therefore, the stoichiometric air-fuel ratio of Neem and Karanja oil methyl esters will be lower than fossil diesel. The Neem oil methyl ester contains 10.77% of O_2 and Karanja oil methyl ester contains 11.02% of O_2 as a result the combustion efficiency of the Karanja oil methyl ester is more when compared to Neem oil methyl ester.

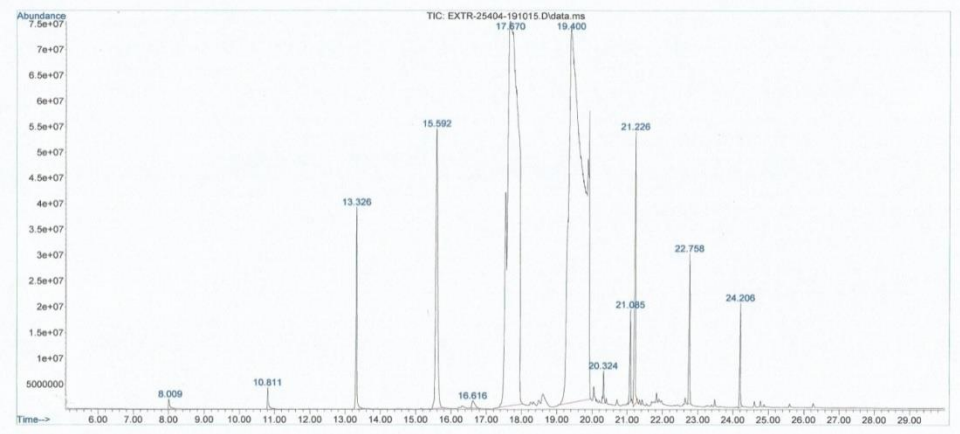

Figure 5 : GC- 5973 N MSD Analysis for Methyl Ester of Neem Oil

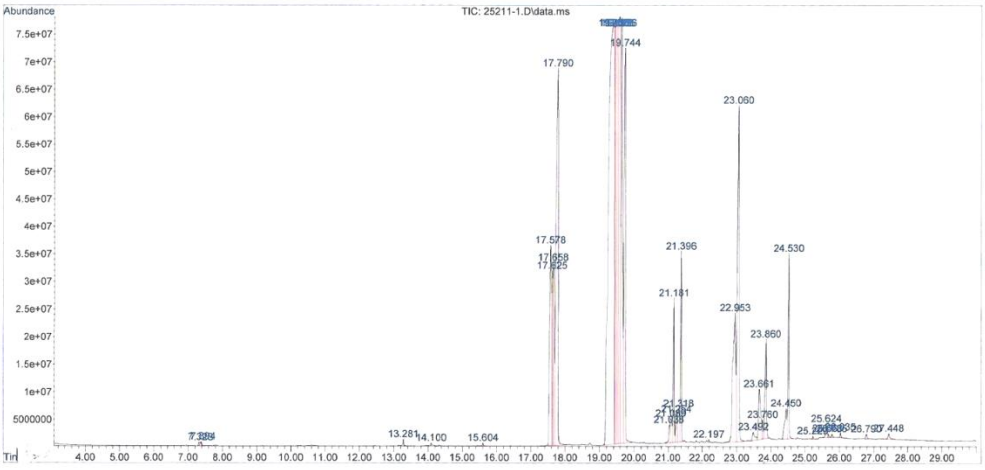

Figure-6 : GC- 5973 N MSD Analysis for Methyl Ester of Karanja Oil

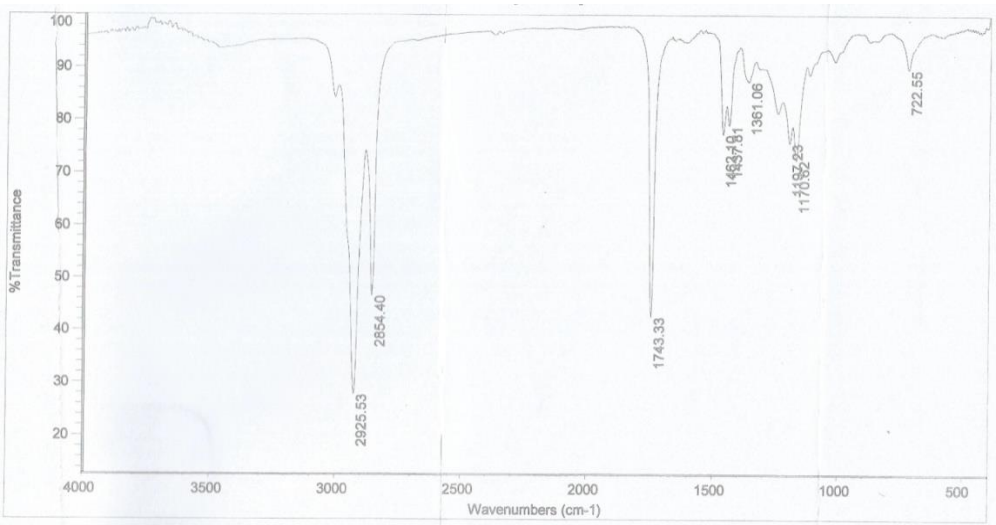

Figure 7: Infrared Spectra of Neem oil Methyl Ester

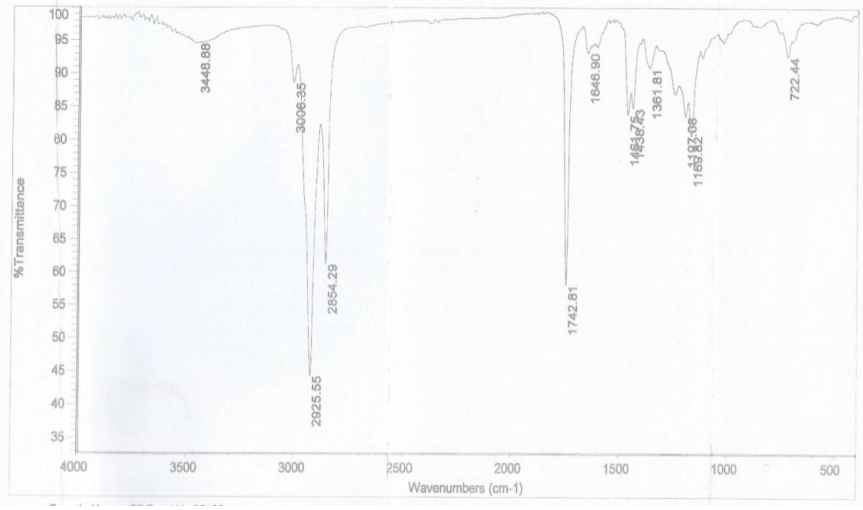

Figure 8: Infrared Spectra of Karanja oil Methyl Ester

Table 1: Elemental composition of Neem and Karanja oil Methyl Esters

S. No	Element	Neem Methyl Ester (Wt.%)	Karanja Methyl Ester (Wt.%)
1	Carbon	75.87	76.12
2	Hydrogen	12.55	12.00
3	Nitrogen	0.24	NF
4	Oxygen	10.77	11.02
5	Sulfur	--	NF
6	C/H	6.04	6.34

Table 2: Comparison of Elemental composition and 'C/H' Ratio with Petro diesel and Biodiesel

Element (Wt. %)	Petro Diesel	Karanja Methyl Ester (Wt.%)	Neem Methyl Ester (Wt.%)	ASTM D6751-02 (Bio-diesel)
Carbon (C)	86.25	76.12	75.87	77
Hydrogen (H)	12.5	12.00	12.55	12
Nitrogen (N)	0	NF	0.24	-----
Sulfur (S)	0.25	NF	--	0.05
Oxygen (O_2)	1.0	11.02	10.77	11
C/H ratio	6.9	6.34	6.04	-----

Table 3: Physical and Chemical properties of test fuels in comparison to some ASTM biodiesel standards

Property	Units	Petro Diesel	Neem methyl ester	Karanja methyl ester	ASTM D6751-02 (Biodiesel)
Carbon chain	C_n	HC C_8-C_{32}	FAME $C_{8\text{-}25}$	FAME C_{16} – C_{24}	FAME C_{12} – C_{22}
Density @30 °C D93	kg/m^3	820	884	878	870-900
Lower calorific value	kJ/kg	42500	37500	38300	-----
Kinematic viscosity @40 °C D445	cSt	2.25	5.77	4.2	1.9-6.0
Cetane Number D613	----	48	50	55.8	47 min
Iodine Value DIN53241	Iodine/100g	38	90	81	120 max
Flash point, Closed cup D93	°C	66	158	175	130
Fire point	°C	98	162	179	-----
Molecular Weight	g/mol	226	270	281	--

3.3.2 Hydrocarbons

Hydrocarbons containing between six and 10 carbon molecules are the top components of most fuels, regardless of whether they are alkanes, alkenes, or cyclic. In general, these molecules are burned to produce energy. The Neem methyl ester contains 75.87 % Carbon and 12.55 % hydrogen and for Karanja methyl ester 76.12% Carbon and 12% hydrogen are present as shown in Table 1.

Burning hydrocarbons requires oxygen. The hydrocarbon and oxygen combine, in a process called combustion, to produce water, carbon dioxide, and energy. Of course, these molecules are not the only products of the combustion of hydrocarbon. Hydrocarbons that are contaminated with atoms such as sulfur and nitrogen will also produce nitrogen dioxide and sulfur dioxide [12, 13].

Because hydrocarbons are composed purely of carbon and hydrogen, their combustion with oxygen can only produce water as a result of the combination between hydrogen and oxygen and carbon dioxide as a result of the combination of carbon and oxygen. The energy produced by burning a hydrocarbon comes from breaking both carbon-hydrogen and carbon-carbon bonds and recombining them into carbon-oxygen and hydrogen-oxygen bonds. Because an unsaturated hydrocarbon has fewer hydrogen carbon bonds, it has less hydrogen per molecule than a similar unsaturated hydrocarbon and will produce more carbon dioxide. This also means unsaturated hydrocarbons produce less energy when burned than do saturated hydrocarbons. In order to gain the same amount of energy, a greater quantity of unsaturated hydrocarbon must be burned and as a result more carbon dioxide is created in the process [14-16]. Thus, unsaturated hydrocarbons are less environmentally friendly than saturated hydrocarbons. Table 2 shows comparison of Elemental composition and 'C/H' Ratio with Petro diesel and biodiesel.

3.4 Fourier Transform Infrared Spectroscopy (FTIR) analysis

FT-IR is used to determine the different functional group such as alcohol, alkane, alkynes, alkenes and other such groups present in the substance which here is neem oil methyl ester and karanja oil methyl ester. Interpreting infrared (IR) spectra is of immense help to structure determination. Not only will it tell you what functional groups and structural elements are there, it will also clarify which ones are not there, and also concentration of bands by using values of transmittance [17].In most chemical environment, the hydroxyl group does not exist in insolation and a high degree of association is experienced as a result of extensive hydrogen bonding with other hydrogen groups [17].

The special contributions are characterized as C-H stretching and bending vibrations which are unique for each sample. Is the main absorption below 3000 cm^{-1}, If so, the compound is probably aliphatic. If the main absorptions are approximately 2935 and 2860 cm^{-1} and there are also absorptions at 1470 and 720cm^{-1}, then the compound probably contains a long linear aliphatic chain.

Stretching absorptions of a vibrating chemical bond occur at higher frequencies (wave number) than the corresponding bending or bond deformation vibrations, with the perception of course that energy and frequency are proportionally related. A good example is the C=H set of vibrations, observed in the hydrocarbon spectra and in virtually all organic compounds. Here, the sample C=H stretching vibrations for saturated aliphatic species occur between 3000 and 2800 cm^{-1} and the corresponding simple bending vibrations nominally occur between 1500 and 1300 cm^{-1}.

It could be concluded that the most abundant type of bond on both neem oil methyl ester and karanja oil methyl ester are C-H, C-O, O-H and C-N. The modes of the bonds are stretch, bending and rocking. If you observe the report of Neem and Karanja oils methyl esters, one can notice the major difference between them is –C=C- that is observed at 1646.90 cm^{-1}. Due to this bond we can say that Karanja oil Methyl Ester has more calorific value in comparison with Neem oil Methyl Ester (Figure 7 & 8).

3.5 Comparison of Biofuels with ASTM biodiesel standards

For selecting a good Fatty Acid Methyl Ester one has to select a ester which has high cetane number, less density and viscous, moderate flash and fire point that can be easily achieved in the combustion engine [18]. Therefore from the comparison Table 3, Karanja oil Methyl Ester is much better when compared with Neem oil Methyl Ester [19].

4. CONCLUSION

The production of methyl ester from Karanja and Neem oils offers a triple-facet solution: economic, environmental, eco friendly. The best conditions of operation for Neem oil methyl ester is at 9:1 alcohol molar ratio to oil with 1.5% KOH catalyst,65^{o}C reaction temperature and time 120 min gives 90% optimum yield. For Karanja oil methyl ester the methanol molar ratio to oil is 9:1 with catalyst 1 wt% at 55^{o} C for 90 minutes gives optimum yield of 92 %. Therefore Karanja oil methyl ester (92%) was better than Neem oil methyl ester (90%) as it requires less catalyst, temperature and reaction time. By the reports of GC-MS, Neem oil Methyl Ester was more stable than Karanja oil Methyl Ester as it has more saturated methyl ester but the calorific value was more in Karanja oil methyl ester due to more unsaturated methyl esters present thereby increasing the cetane number. From the elemental analysis report, sulphur and nitrogen were not present in Karanja oil Methyl ester whereas in Neem oil Methyl Ester little amount of nitrogen was present and C/H ratio was high in Karanja oil Methyl Ester. With the help of FTIR analysis we can find the strength of the bonds present. The bonds such as C-H, C-O, O-H and C-N were abundant in Neem oil Methyl Ester as well as Karanja oil Metyl Esters. The modes of the bonds are stretch, bending and rocking. The major difference

between them was Alkenes present in Karanja oil Metyl Ester which gives high energy. Thus Karanja oil was more suitable than Neem oil for the production of methyl ester.

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